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(74) Agents: CROSBY, Margaret, M. et al.; Bullwinkel Partners, Ltd., 19 South LaSalle Street, #1300, Chicago, IL 60603 (US).

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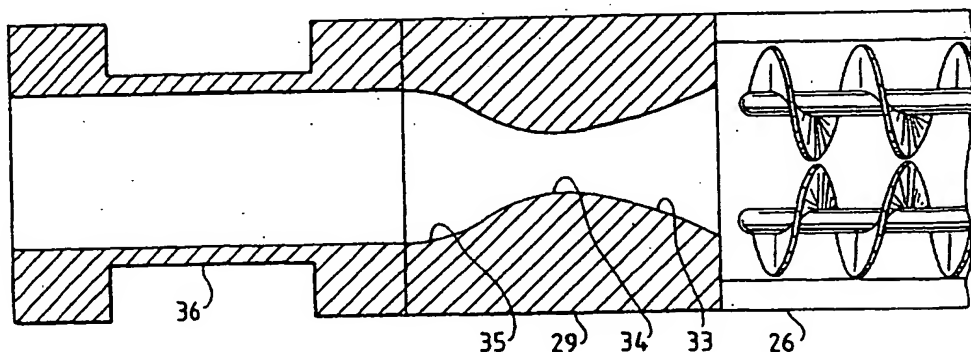
(71) Applicant: U.S. PLASTIC LUMBER IP CORP.
[US/US]; Suite 440 West, 2300 Glades Road, Boca Raton, FL 33431 (US).

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(72) Inventors: DAHL, Michael, E.; 424 Woodside Avenue, Hinsdale, IL 60521 (US). ROTTINGHAUS, Robert, G.; 929 Division Street, Lisle, IL 60532 (US). STEPHENS, Andrew, H.; 323 S. Lincoln Street, Hinsdale, IL 60521 (US).

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(54) Title: EXTRUDED WOOD POLYMER COMPOSITE AND METHOD OF MANUFACTURE



(57) Abstract: An extruded composite artificial lumber product is manufactured from wood fibers, a polyethylene matrix and a foaming agent. A mixture is extruded through a molding die which forms the profile of the desired product. The endothermic foaming agent causes greater expansion in the center of the extruded profile and increased density at the outer edges of the extruded profile.



WO 00/46010 A1

TITLE

3 EXTRUDED WOOD POLYMER COMPOSITE
4 AND METHOD OF MANUFACTURE

6 SPECIFICATION

7 Be it known that we, Michael E. Dahl, Robert G.
8 Rottinghaus, and Andrew H. Stephens, have invented
9 certain new and useful improvements in an Extruded Wood
10 Polymer Composite and Method of Manufacture, of which
11 the following is a specification.

12

13 FIELD OF THE INVENTION

14 This invention relates to an extruded composite
15 artificial lumber product manufactured from wood fiber
16 and polyethylene, including recycled polyethylene, and
17 its method of manufacture.

18

19 DESCRIPTION OF THE PRIOR ART

20 The prior art reflects many attempts to make an
21 acceptable artificial lumber out of wood fiber and
22 thermoplastics, particularly using recycled materials.
23 Some, such as the product and process disclosed in Laver
24 U.S. Pat. No. 5,516,472 Extruded Synthetic Wood
25 Composition and Method for Making Same, have enjoyed
26 some commercial utility as being a relatively cost-
27 efficient means of re-using materials, which might

1 otherwise be wasted, in the manufacture of lumber-like
2 products which are relatively strong, dimensionally
3 stable and moisture-resistant. Laver teaches that a
4 cellulosic wood fiber material may be mixed with a
5 thermoplastic material and a cross-linking material, all
6 of which are subject to heat (about 180 ° C) and
7 pressure in a twin-screw extruder until they become
8 plastic. The plastic mixture is then extruded through a
9 series of dies including a "stranding" die having
10 multiple orifices in a honeycomb pattern to orient the
11 fibers in the plastic material in a longitudinal
12 direction. The die also includes gas evacuation
13 passages to relieve unwanted process gas, such as from
14 volatile cross-linking agents. As a result, according
15 to Laver, a product is created which may be formed into
16 intricate shapes with no expansion after leaving the
17 molding die. A water spray system cools the product
18 after it leaves the extrusion die, leaving a hardened
19 gloss or glaze on the surface of the product.

20 Brandt, et al. US 5,827,462 (10/27/98) discloses an
21 extruded synthetic wood product using a twin screw
22 extruder discharging a plasticized material which is 50-
23 70% cellulosic and 20-40% thermoplastic, containing
24 cross-linking agents into a transition die and then a

1 stranding die, and then cooling the extruded product
2 with water spray.

3 Deaner, et al. US 5,827,607 (10/27/98) discloses a
4 method of using a twin screw extruder to form composite
5 thermoplastic pellets having 45-70% polyvinyl chloride
6 and 30-50% wood fiber (not wood flour), and being at
7 least 0.1 mm long with an aspect ratio of 1.8. After
8 being pelletized, the material is used as feedstock for
9 a three stage extruder in which the pellets are mixed,
10 melted, and then formed at 195-200° C using a wax
11 lubricant, into structural shapes for doors, windows and
12 the like.

13 Brooks, et al. US 5,082,605 (1/21/92) discloses a
14 method for extruding a composite synthetic wood product
15 containing encapsulated cellulosic fibers. The feed
16 mixture contains polyethylene and up to 10-15%
17 polypropylene, in ratios in a general range of 40/60 to
18 60/40 fiber/polymer. The desirable fiber particles are
19 no more than 1.5 inches, and the polymeric materials are
20 ground to particles of no more than 0.25 inches. The
21 fiber particles are encapsulated in a jacketed
22 compounder at 300-600° F. Clumps of encapsulated
23 material no more than 1.5 inches in length are
24 introduced into a jacketed extruder, at temperatures

1 less than 450° F, and extruded through a fiber alignment
2 plate and then a heated forming die.

3 Brooks, et al. US 5,088,910 (2/18/92) discloses a
4 system for making synthetic wood products. Wood fiber
5 is mixed with thermoplastic material, including both
6 LDPE and HDPE, in plastic/fiber ratios of 40/60 to
7 60/40, and then heated and kneaded before being formed
8 into golf-ball sized chunks. A fiber alignment plate is
9 positioned ahead of the final extrusion die. The
10 product is cut to desired length using a flying cutoff
11 knife mounted on a table which tracks the movement of
12 the formed material as it leaves the extruder.

13 Brooks, et al. US 5,759,680 (6/2/98) discloses an
14 extruded fiber/polymer composite material in ratios of
15 40/60 to 60/40. The feed material is heated to a
16 working temperature between 190° and 350° F in a
17 jacketed mixer, until it reaches a clumpy, doughy
18 consistency, after which it goes to a size reduction
19 unit, and finally to a compounding extruder using a
20 fiber alignment plate ahead of the final extrusion die.
21 The disclosure teaches that the feedstock should contain
22 no foaming agent, and all but one of the claims reflects
23 that limitation by being limited to "unfoamed" polymeric
24 material. (The one claim not having that limitation is

1 limited to a process which achieves plasticization in a
2 separate "jacketed mixer" prior to extrusion, which
3 makes the process entirely different from the present
4 invention.)

5 SUMMARY OF THE INVENTION

6 It is a primary general object of the present
7 invention to provide a superior extruded wood polymer
8 composite and method of manufacture which is easier,
9 cheaper and quicker to manufacture, and requires less
10 complex manufacturing steps and equipment.

11 A related general object of the invention is to
12 provide a method which will produce a product which has
13 physical properties as good or better than exhibited by
14 prior art products of a similar kind.

15 A specific object of the invention is to provide a
16 method for manufacturing a superior product which has a
17 lower overall density and specific gravity compared to
18 the prior art, while maintaining all or substantially
19 all of its surface strength, hardness and finish, and
20 moisture resistance. In particular, it is an object to
21 provide an extruded artificial lumber product with
22 similar surface qualities of density, hardness and
23 strength, as the prior art, but having selectively
24 reduced density at its central core. By this means the

1 product of the invention is substantially just as strong
2 as the prior art, but is significantly less dense and
3 more economical to manufacture, and is equal to or
4 superior to the prior art in terms of workability in
5 sawing, drilling, nailing, stapling, and the like.

6 By the method of the present invention, a high-
7 quality wood-like extruded artificial lumber product is
8 produced by finely dividing wood fiber and polyethylene
9 into particles, and then mechanically mixing them
10 together with a measured amount of a powdered
11 endothermic foaming or blowing agent. The resulting
12 feed mix is directly introduced, without pre-
13 pelletization, into a conventional twin-screw extruder
14 where it is compressed and heated into a homogenous
15 plastic state, and then extruded through a molding die
16 to form the structural profile of the desired product.
17 Gases, consisting of vaporized moisture from the
18 feedstock and excess process gas from the foaming agent,
19 is removed by vacuum through passages in the extruder
20 ahead of the molding die. In the process, the carefully
21 controlled amount of foaming agent ingredient has the
22 desirable effect of reducing the density at the center
23 of the extruded profile, while allowing the outer
24 surfaces to remain dense, hard and strong. This has the

1 overall desirable effect of producing a product which is
2 relatively stronger with respect to its density, while
3 continuing to present a smooth, hard well-finished
4 external appearance.

5 It is believed that the controlled amount of
6 foaming agent causes a greater degree of expansion in
7 the center of the extruded profile than at its
8 perimeter, thereby compressing a greater proportion of
9 plastic material against the sides of the extrusion die.
10 This has the effect of increasing the density and
11 strength on the outside of the extrusion, while reducing
12 the density (with no significant loss of overall
13 strength) on the inside. The resulting extruded
14 artificial lumber product can be selectively made with a
15 specific gravity of 1.0, plus or minus 20%, with no
16 significant variation in external dimensions after
17 cooling.

18

19 THE DRAWINGS

20 FIG. 1 is a perspective view of four extruded
21 artificial products, of which one represents a typical
22 prior art product for comparison purposes, and three
23 have been manufactured according to the present
24 invention;

1 FIG. 2 is a schematic diagram of a process
2 embodying the method of the present invention;

3 FIG. 3 is an enlarged horizontal cross-section of
4 the forming die and stabilizing die which receives the
5 molten exudate from the extruder; and

6 FIG. 4 is an enlarged vertical cross-section of the
7 forming die and stabilizing die of Fig. 3.

8

9 DETAILED DESCRIPTION OF THE INVENTION

10 Turning to the drawings, there is shown in Fig. 1 a
11 typical prior art extruded lumber product 10, such as
12 might be manufactured using the process taught in the
13 Laver U.S. Pat. No. 5,516,472. The product 10 might
14 typically be produced in ten foot lengths, with
15 dimensions of 6 inches by 5/4 inches (nominal) and 10,
16 12 or 16 feet in length. This product finds great
17 utility in outdoor benches, tables, and railings, and as
18 deck planking for exterior porches exposed to the
19 weather year-round. Such a prior art product might
20 typically be composed of about two parts finely divided
21 wood fiber and one part finely divided recycled
22 thermoplastic material, along with a lesser amount of
23 thermosetting plastic material. The finely divided
24 ingredients can be mixed directly prior to introduction

1 into an extruder, or they can be pre-pelletized, in the
2 method taught by Deaner, et al. US Patent 5,827,607.
3 Typically, a multiple-stage molding die having a fiber
4 alignment plate or stranding die is used, which aligns
5 the wood fibers, but also cause a high level of back
6 pressure in the extruder.

7 Such prior art artificial lumber planking, while
8 not generally as strong as natural wood, exhibits other
9 favorable qualities. It is generally maintenance free,
10 and can be exposed to the elements indefinitely without
11 significant degradation of either appearance or
12 strength. As for ease of fabrication, it is quite
13 similar to wood in that it can be drilled, sawed, and
14 nailed, and can receive screw and other fasteners, with
15 results very similar to natural wood.

16 However, despite the advantages set forth above,
17 prior art artificial lumber products such as the
18 illustrated example 10 often exhibit deficiencies which
19 can seriously and adversely affect their utility and
20 longevity in certain applications. For example, it has
21 been found that extruded composite products manufactured
22 using the stranding die technology taught in the Laver
23 U.S. Pat. No. 5,516,472 will sometimes suffer from
24 moisture absorption, possibly as a result of having a

1 lower thermoplastic content together with the presence
2 microscopic longitudinal channels created by the forced
3 alignment of the wood fibers during the extrusion
4 process. As a result, the product has, in effect, an
5 "end grain" through which moisture can enter, causing
6 eventually swelling, warping and distortion which can
7 upset the dimensional stability of any structure
8 manufactured with these materials.

9 In addition, while the prior art extruded
10 artificial lumber products 10 generally have a superior
11 surface in terms of strength, hardness and appearance,
12 they are generally quite dense, with some having
13 specific gravities substantially higher than 1.0,
14 meaning that they consume more raw materials per board
15 foot of product, and have a poorer strength-to-weight
16 ratio in comparison to natural wood. They will not
17 float at all.

18 Finally, the manufacture of prior art artificial
19 lumber products 10 by the prior art methods described
20 above is relatively costly and time-consuming because of
21 the need for either pre-pelletization or a pre-melt step
22 in some cases, and for multiple-part extrusion dies
23 (including stranding dies) in others.

1 Referring again to the drawings, there are also
2 shown in Fig. 1 three additional extruded artificial
3 lumber sections 12, 14 and 16, in the form of deck
4 planks, manufactured according to the present invention.
5 Improved plank 12 exhibits the same hard, strong, smooth
6 surface as prior art plank 10, but has at its center a
7 region 13 of reduced density which lowers the overall
8 density and weight of the plank without significantly
9 affecting its strength. Even though the density
10 reduction may reduce the tensile strength and modulus of
11 the product at its center, the fact that the outer
12 surfaces are effectively unaffected causes the overall
13 strength and modulus of the product to be substantially
14 unchanged.

15 The density reduction of plank 12 at its center 13
16 is achieved by the addition of a controlled quantity of
17 foaming agent, preferably up to 1% of an endothermic
18 foaming agent such as bicarbonate of soda. This agent
19 is added and mixed into the wood fiber and thermoplastic
20 polymer components which, together with small quantities
21 of certain other components, comprise the feedstock of
22 the extruder. It has been found that it is possible to
23 control the expansion of the foaming agent in a way
24 which substantially confines it to the center of the

1 extruded product, thereby achieving additional lightness
2 without any sacrifice in surface characteristics or
3 overall strength.

4 The amount of endothermic foaming agent in the
5 feedstock mix has been found to be relatively critical.
6 Referring again to Fig. 1, plank 14 exhibits bowed outer
7 surfaces because of excessive expansion at its center
8 15. Similarly, the center 17 of plank 16 has not
9 expanded sufficiently, or has even shrunk after leaving
10 the extruder, giving the cross-section a "dog bone"
11 shape which is also unacceptable. It is therefore
12 important to adjust and balance the concentration of
13 endothermic foaming agent against the wood fiber and
14 thermoplastic polymer components of the feedstock
15 mixture so that a plank 12 with dimensionally stable
16 surfaces is achieved, and not a bowed plank 14 or sunken
17 plank 16 which may possess a reduced density at its
18 center, but which may be dimensionally unacceptable.

19 Turning to Fig. 2, there is shown in schematic form
20 a production line for producing the improved,
21 dimensionally stable plank 12 of the present invention.
22 A supply of wood fiber or other fibrous cellulosic
23 material 18 is introduced into a pulverizer or shredder
24 19 where it is finely divided into particles having a

1 maximum length dimension generally no smaller than 80
2 mesh (about 0.007 inches), and no larger than about 0.5
3 inches, with the preferred range being 10-40 mesh.
4 Another supply of thermoplastic material 20, which is
5 preferably scrap polyethylene such as may be reclaimed
6 from a materials recycling program, is similarly finely
7 divided in a pulverizer or shredder 21 into particles
8 generally no smaller than 80 mesh, with the preferred
9 range being 10-60 mesh.

10 After pulverization, the finely divided wood fiber
11 and thermoplastic particles are conveyed, such as by air
12 conveyor, to a mixer 22. To the mixer 22 is also added
13 a quantity of powdered endothermic foaming agent 23 such
14 as bicarbonate of soda, and (if desired) up to about 1%
15 of a wax lubricant 24.

16 In practice, the following ranges (parts by weight)
17 of components have been found most desirable in
18 achieving the objects of the invention:

		Wood Fiber	Polymer	Foaming Agent	Lubricant
21	Composition A	50	50	0.6	0.8
22	Composition B	60	40	0.3	1.0
23	Composition C	40	60	0.7	0.6

1 If desired, up to 5 parts of a thermoplastic olefin
2 can also be added for optimizing melt flow
3 characteristics.

4 According to the invention, the wood fiber,
5 thermoplastic and foaming agent ingredients are
6 thoroughly mixed in the mixer 22 and then conveyed, by
7 means such as an air conveyor, to the input hopper 25 of
8 a screw-type extruder 26. Excellent results have been
9 achieved using the commercially available Cincinnati
10 Milacron CM-80-BP twin screw extruder driven by motor
11 27. As is well known in the art, the twin screw
12 extruder uses meshed counter-rotating flights (not
13 shown) which have a larger pitch at the inlet end and a
14 smaller pitch at the output end. The flights are heated
15 internally, and the extruder barrel is also heated.

16 In combination, the heat imparted to the feedstock
17 mixture by the heated extruder flights and barrel, plus
18 the mechanical shearing and compression caused by the
19 differential pitch of the flights, cause the feedstock
20 mixture temperature to be raised to a point where it
21 becomes plastic and homogenous, with the wood fibers
22 being intimately mixed, coated and bound in the melted
23 thermoplastic. In addition, any residual moisture in
24 the feedstock components is vaporized, and as the

1 mixture heats further, its temperature is desirably in
2 the range of 320° F to 400° F, which causes the
3 endothermic foaming agent to become activated, absorbing
4 some of the heat energy and releasing carbon dioxide
5 gas.

6 As the heated and compressed feedstock approaches
7 the extruder die 29 at the exit end of the extruder,
8 excess volatiles including vaporized moisture and excess
9 foaming agent gas (principally carbon dioxide) are
10 removed from the extruder ahead of the molding die by a
11 vacuum pump 28. In practice, it has been found that the
12 best results are obtained at vacuum levels of at least
13 25 inches of mercury, with the best operating range
14 being between 27 and 30 inches of mercury. With less
15 vacuum, the resulting product is more sensitive to
16 moisture, possibly because the remaining volatiles
17 (water and carbon dioxide) permeating the melt and
18 create fissures in the final product, into which water
19 may penetrate. On the other hand, vacuum levels of 30
20 inches of mercury and more tend to negate the effect of
21 the foaming agent, leading to insufficient density
22 reduction, insufficient dimensional stability on leaving
23 the extruder, and poor workability in the finished
24 product.

1 With the process of the present invention, no
2 special multiple die sets, and no fiber alignment or
3 stranding die, are needed to produce a strong, stable,
4 moisture-resistant product. As shown in Figs. 3 and 4,
5 the extrusion die 29 has a converging entrance 33
6 leading to a throat 34 sized to produce the desired
7 degree of pressure drop leaving the extruder, and a
8 diverging exit 35 passage allowing for expansion of the
9 melt in cross-section to form the desired profile of the
10 extruded product.

11 From the exit passage the extruded product passes
12 through a stabilization die 36 where it cools
13 sufficiently to retain its shape upon entering the spray
14 chamber 30. In practice, the extruded material leaving
15 the throat of the die expands just sufficiently to take
16 the fill the exit passage and thereby take its final
17 shape, without undue pulling or dragging across its
18 surface which might cause fissures known as "melt
19 fractures".

20 From the extruder 26 and die 29, the formed ribbon
21 of extruded product passes into a spray chamber 30 where
22 it is cooled by spray jets of water from a reservoir 31
23 as is well understood in the art. Once cooled, it
24 passes by conventional means to a cutoff station 32

1 where a traveling table or "flying" cutoff knife or saw
2 cuts the product to any length desired.

3 A typical product manufactured by the method of the
4 invention has been found to exhibit the following
5 characteristics (typical values):

6	Modulus of elasticity	285,758 psi	ASTM D4761
7	Modulus of rupture	1676 psi	ASTM D4761
8	Tensile strength	786 psi	ASTM D198
9	Shear strength	706 psi	ASTM D143
10	Screw withdrawal force	650 lb/in	ASTM D1761
11	Nail withdrawal force	177 lb/in	ASTM D1761
12	Coefficient of thermal expansion	4.5×10^{-5}	ASTM E228
13	Water absorption	1.66%	ASTM D1037
14	Density (S.G.)	1.0	

1 I CLAIM AS MY INVENTION:

2 1. A process for manufacturing a composite
3 extruded structural product having a desired profile
4 from thermoplastic material and wood fiber comprising
5 the steps of:

6 finely dividing the thermoplastic material and wood
7 fiber each into particles no smaller than about 0.007
8 inches and no larger than about 0.5 inches in length;
9 mechanically mixing together the thermoplastic
10 particles and the wood fiber particles in a ratio of
11 between 60%-40% and 40%-60% by weight, together with an
12 effective amount of a foaming agent, to form a feedstock
13 mixture;

14 introducing the feedstock mixture, without pre-
15 pelletization, into a screw-type extruder;

16 mechanically mixing, compressing and heating said
17 feedstock mixture in said extruder until it becomes
18 plastic and homogenous;

19 extruding said heated, plastic, homogenous mixture
20 through a molding die into the structural profile of a
21 desired product;

22 cooling said extruded product upon emerging from
23 said molding die; and

1 cutting the cooled extruded product into desired
2 lengths.

3 2. The process of claim 1 in which an effective
4 amount of foaming agent ingredient is selected to create
5 an extruded product having a specific gravity of between
6 about 0.8 and about 1.2 with no significant dimensional
7 variation after cooling.

8 3. The process of claim 1 in which the effective
9 amount of foaming agent ingredient is up to about 1% by
10 weight.

11 4. The process of claim 1 in which the foaming
12 agent ingredient is an endothermic foaming agent.

13 5. The process of claim 1 in which the foaming
14 agent ingredient is bicarbonate of soda.

15 6. The process of claim 1 including the
16 additional step of extracting excess volatiles under
17 vacuum from said extruder, thereby producing an extruded
18 product having a surface which is relatively dense,
19 tight-grained and strong, and a center which is
20 relatively more porous and less dense.

21 7. The process of claim 6 in which the vacuum
22 extraction step is performed using a vacuum of at least
23 25 inches of mercury.

1 8. The process of claim 1 in which up to 1% by
2 weight of wax lubricant is mixed into the feedstock
3 mixture prior to introduction into the extruder.

4 9. The process of claim 1 in which up to 5% by
5 weight of thermoplastic olefin is mixed into the
6 feedstock mixture prior to introduction into the
7 extruder.

8 10. The process of claim 1 in which the molding
9 die has a converging entrance, a throat, and a diverging
10 exit terminating in the profile of the desired
11 structural product.

12 11. The process of claim 1 in which the extruded
13 product upon emerging from said molding die is cooled
14 with a direct water spray, and said cooled extruded
15 product is cut into desired lengths with a traveling
16 saw.

17 12. A process for manufacturing a composite
18 extruded structural product having a desired profile
19 from recycled polyethylene and wood fiber comprising the
20 steps of:

21 finely dividing recycled polyethylene and wood
22 fiber each into particles of a size between 10 mesh and
23 40 mesh;

1 mechanically mixing together the polyethylene
2 particles and the wood fiber particles in a ratio of
3 between 60%-40% and 40%-60% by weight, and an effective
4 amount of a powdered endothermic foaming agent, to form
5 a feedstock mixture;

6 introducing the feedstock mixture, without pre-
7 pelletization, into a heated screw-type extruder
8 discharging into a molding die, said molding die having
9 an entrance, a throat, and an exit having the shape of a
10 desired product;

11 mechanically mixing, compressing and heating said
12 feedstock mixture in said extruder until it becomes
13 plastic and homogenous;

14 extracting excess volatiles and foaming agent
15 process gas under vacuum from said feedstock mixture
16 prior to entering said molding die;

17 forcing said heated, plastic, homogenous mixture
18 through said molding die to produce an extruded product
19 having a surface which is relatively dense, tight-
20 grained and strong, and a center which is relatively
21 more porous and less dense;

22 cooling said extruded product upon emerging from
23 said molding die; and

1 cutting the cooled extruded product into desired
2 lengths.

3 13. A composite extruded artificial lumber product
4 having a surface which is relatively dense, tight-
5 grained and strong, and a center which is relatively
6 more porous and less dense, manufactured by the process
7 of claim 1.

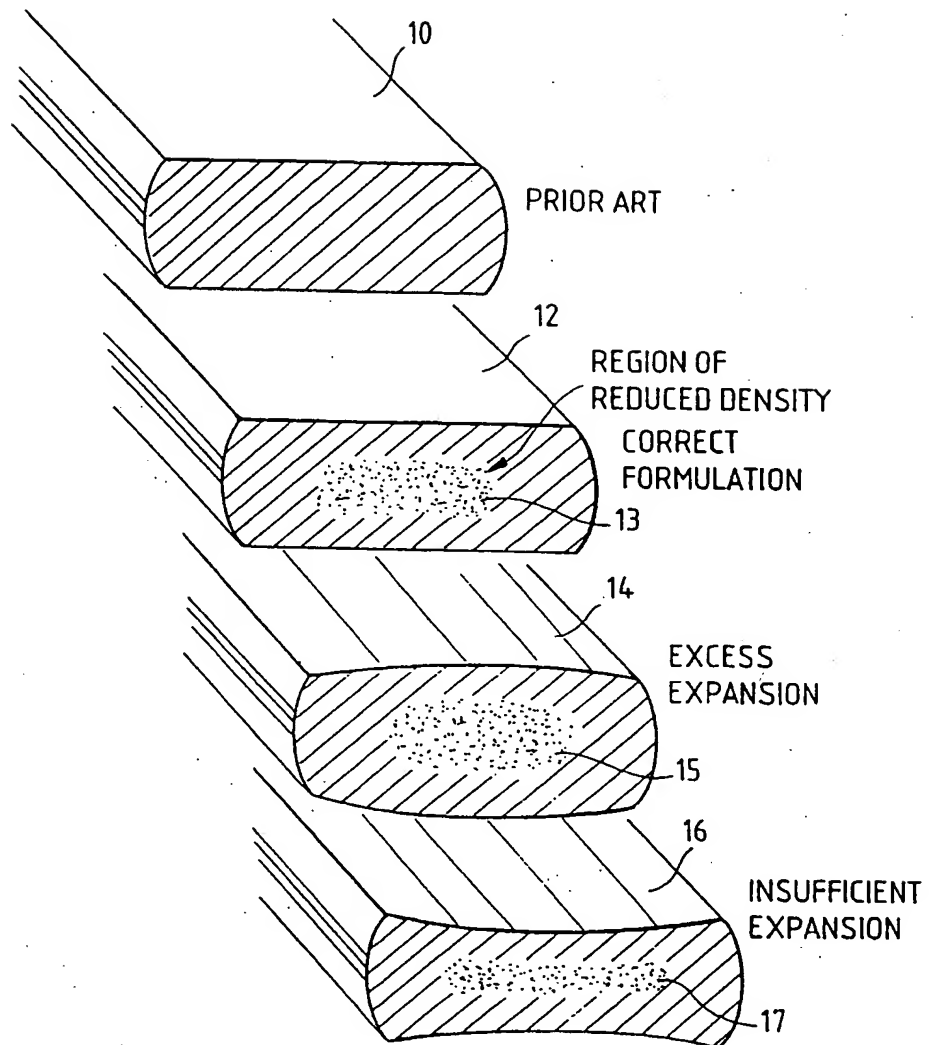
8 14. A composite extruded artificial lumber product
9 having a surface which is relatively dense, tight-
10 grained and strong, and a center which is relatively
11 more porous and less dense, manufactured by the process
12 of claim 12.

13 15. The composite extruded artificial lumber
14 product of claim 13 having a specific gravity between
15 about 0.8 and about 1.2 with no significant dimensional
16 variation after cooling.

17 16. The composite extruded artificial lumber
18 product of claim 14 having a specific gravity between
19 about 0.8 and about 1.2 with no significant dimensional
20 variation after cooling.

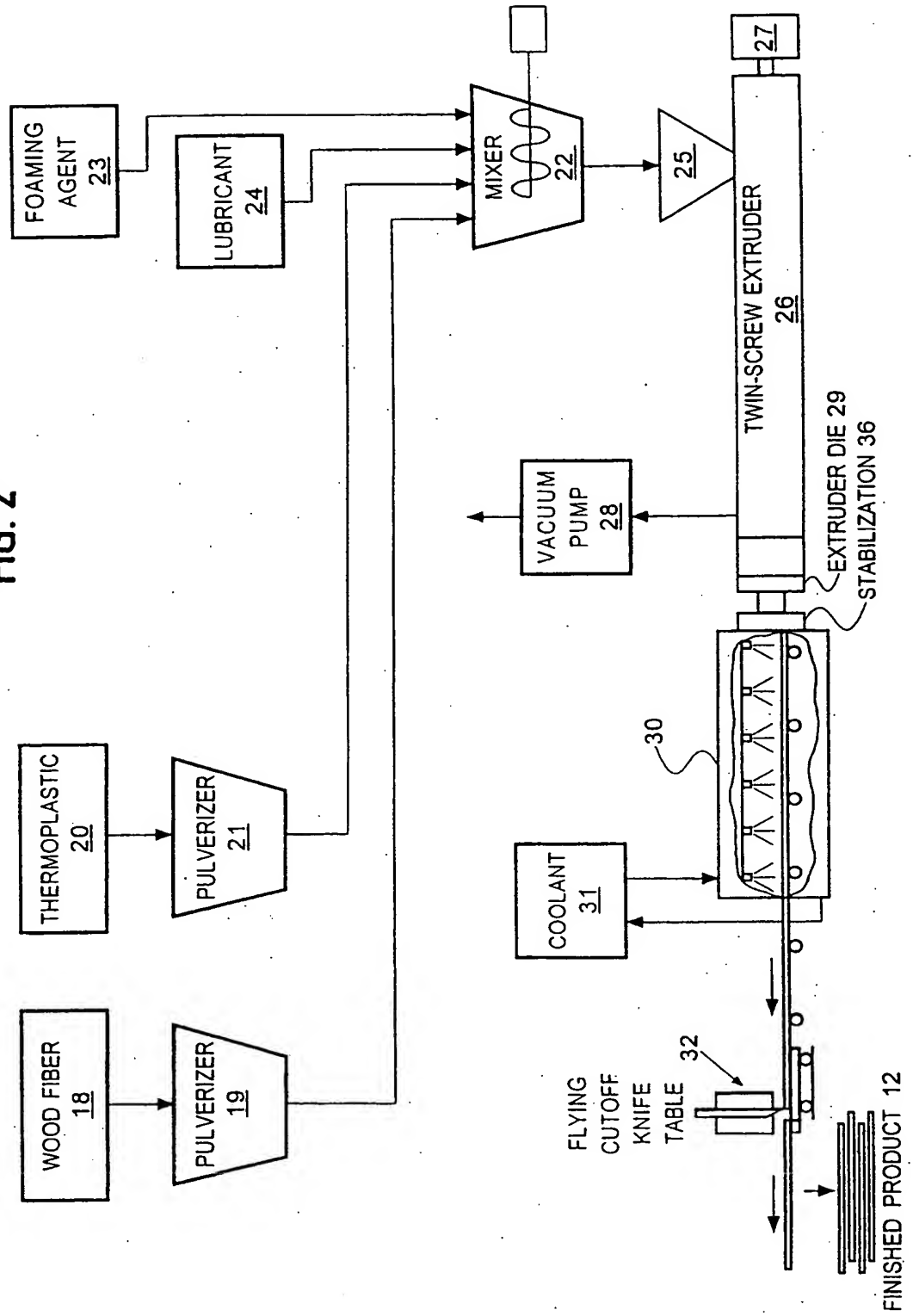
1/3

FIG. 1



2/3

FIG. 2



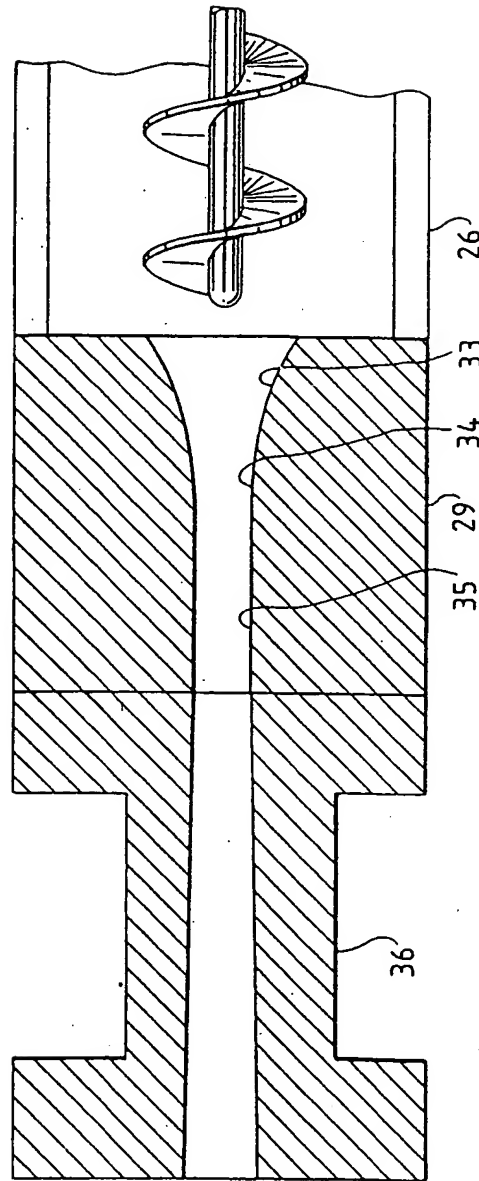


FIG. 3

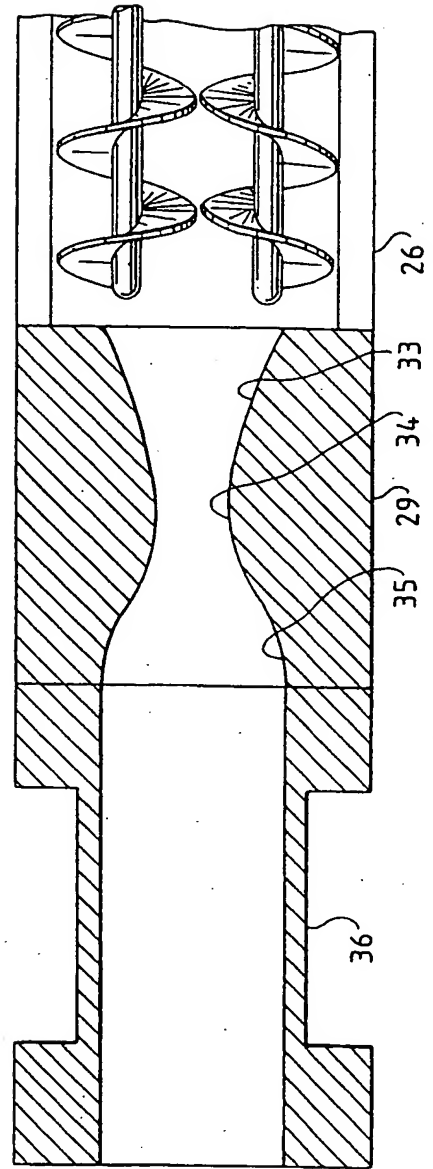


FIG. 4

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/02345

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) :B29C 47/78, 47/36

US CL : 264/118,122,913,920; 428/903.3

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 264/118,122,913,920; 428/903.3; 425/382R, 382.4

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WEST 2.0 search terms, see claim 1

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5,516,472 A (LAVER et al) 14 May 1996	1-16

☐ Further documents are listed in the continuation of Box C.☐ See patent family annex.

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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Date of the actual completion of the international search

05 JUNE 2000

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13 JUN 2000

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RICH WEISBERGER

Telephone No. (703) 308-2351

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(74) Agents: CROSBY, Margaret, M. et al.; Bullwinkel Partners, Ltd., 19 South LaSalle Street, #1300, Chicago, IL 60603 (US).

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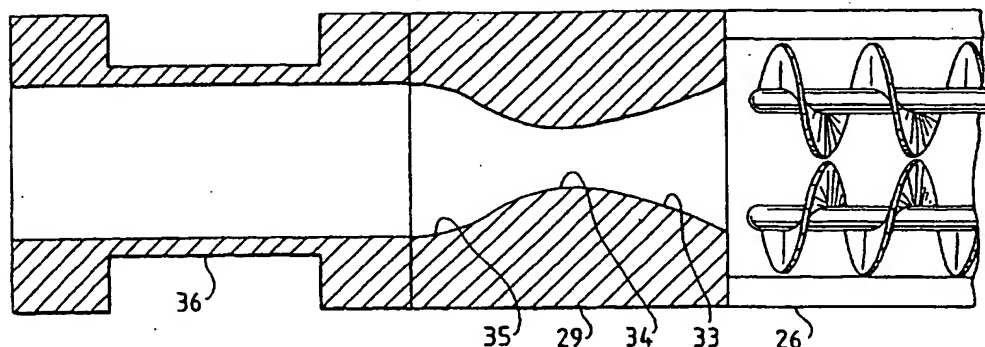
(71) Applicant: U.S. PLASTIC LUMBER IP CORP.
[US/US]; Suite 440 West, 2300 Glades Road, Boca Raton, FL 33431 (US).

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see PCT Gazette No. 41/2001 of 11 October 2001, Section II

(72) Inventors: DAHL, Michael, E.; 424 Woodside Avenue, Hinsdale, IL 60521 (US). ROTTINGHAUS, Robert, G.; 929 Division Street, Lisle, IL 60532 (US). STEPHENS, Andrew, H.; 323 S. Lincoln Street, Hinsdale, IL 60521 (US).

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: EXTRUDED WOOD POLYMER COMPOSITE AND METHOD OF MANUFACTURE



(57) Abstract: An extruded composite artificial lumber product is manufactured from wood fibers, a polyethylene matrix and a foaming agent. A mixture is extruded through a molding die which forms the profile of the desired product. The endothermic foaming agent causes greater expansion in the center of the extruded profile and increased density at the outer edges of the extruded profile.

WO 00/46010 A1

TITLE

3 EXTRUDED WOOD POLYMER COMPOSITE
4 AND METHOD OF MANUFACTURE
5

6 SPECIFICATION

7 Be it known that we, Michael E. Dahl, Robert G.
8 Rottinghaus, and Andrew H. Stephens, have invented
9 certain new and useful improvements in an Extruded Wood
10 Polymer Composite and Method of Manufacture, of which
11 the following is a specification.
12

13 FIELD OF THE INVENTION

14 This invention relates to an extruded composite
15 artificial lumber product manufactured from wood fiber
16 and polyethylene, including recycled polyethylene, and
17 its method of manufacture.
18

19 DESCRIPTION OF THE PRIOR ART

20 The prior art reflects many attempts to make an
21 acceptable artificial lumber out of wood fiber and
22 thermoplastics, particularly using recycled materials.
23 Some, such as the product and process disclosed in Laver
24 U.S. Pat. No. 5,516,472 Extruded Synthetic Wood
25 Composition and Method for Making Same, have enjoyed
26 some commercial utility as being a relatively cost-
27 efficient means of re-using materials, which might

1 otherwise be wasted, in the manufacture of lumber-like
2 products which are relatively strong, dimensionally
3 stable and moisture-resistant. Laver teaches that a
4 cellulosic wood fiber material may be mixed with a
5 thermoplastic material and a cross-linking material, all
6 of which are subject to heat (about 180 ° C) and
7 pressure in a twin-screw extruder until they become
8 plastic. The plastic mixture is then extruded through a
9 series of dies including a "stranding" die having
10 multiple orifices in a honeycomb pattern to orient the
11 fibers in the plastic material in a longitudinal
12 direction. The die also includes gas evacuation
13 passages to relieve unwanted process gas, such as from
14 volatile cross-linking agents. As a result, according
15 to Laver, a product is created which may be formed into
16 intricate shapes with no expansion after leaving the
17 molding die. A water spray system cools the product
18 after it leaves the extrusion die, leaving a hardened
19 gloss or glaze on the surface of the product.

20 Brandt, et al. US 5,827,462 (10/27/98) discloses an
21 extruded synthetic wood product using a twin screw
22 extruder discharging a plasticized material which is 50-
23 70% cellulosic and 20-40% thermoplastic, containing
24 cross-linking agents into a transition die and then a

1 stranding die, and then cooling the extruded product.
2 with water spray.

3 Deaner, et al. US 5,827,607 (10/27/98) discloses a
4 method of using a twin screw extruder to form composite
5 thermoplastic pellets having 45-70% polyvinyl chloride
6 and 30-50% wood fiber (not wood flour), and being at
7 least 0.1 mm long with an aspect ratio of 1.8. After
8 being pelletized, the material is used as feedstock for
9 a three stage extruder in which the pellets are mixed,
10 melted, and then formed at 195-200° C using a wax
11 lubricant, into structural shapes for doors, windows and
12 the like.

13 Brooks, et al. US 5,082,605 (1/21/92) discloses a
14 method for extruding a composite synthetic wood product
15 containing encapsulated cellulosic fibers. The feed
16 mixture contains polyethylene and up to 10-15%
17 polypropylene, in ratios in a general range of 40/60 to
18 60/40 fiber/polymer. The desirable fiber particles are
19 no more than 1.5 inches, and the polymeric materials are
20 ground to particles of no more than 0.25 inches. The
21 fiber particles are encapsulated in a jacketed
22 compounder at 300-600° F. Clumps of encapsulated
23 material no more than 1.5 inches in length are
24 introduced into a jacketed extruder, at temperatures

1 less than 450° F, and extruded through a fiber alignment
2 plate and then a heated forming die.

3 Brooks, et al. US 5,088,910 (2/18/92) discloses a
4 system for making synthetic wood products. Wood fiber
5 is mixed with thermoplastic material, including both
6 LDPE and HDPE, in plastic/fiber ratios of 40/60 to
7 60/40, and then heated and kneaded before being formed
8 into golf-ball sized chunks. A fiber alignment plate is
9 positioned ahead of the final extrusion die. The
10 product is cut to desired length using a flying cutoff
11 knife mounted on a table which tracks the movement of
12 the formed material as it leaves the extruder.

13 Brooks, et al. US 5,759,680 (6/2/98) discloses an
14 extruded fiber/polymer composite material in ratios of
15 40/60 to 60/40. The feed material is heated to a
16 working temperature between 190° and 350° F in a
17 jacketed mixer, until it reaches a clumpy, doughy
18 consistency, after which it goes to a size reduction
19 unit, and finally to a compounding extruder using a
20 fiber alignment plate ahead of the final extrusion die.
21 The disclosure teaches that the feedstock should contain
22 no foaming agent, and all but one of the claims reflects
23 that limitation by being limited to "unfoamed" polymeric
24 material. (The one claim not having that limitation is

1 limited to a process which achieves plasticization in a
2 separate "jacketed mixer" prior to extrusion, which
3 makes the process entirely different from the present
4 invention.)

5 SUMMARY OF THE INVENTION

6 It is a primary general object of the present
7 invention to provide a superior extruded wood polymer
8 composite and method of manufacture which is easier,
9 cheaper and quicker to manufacture, and requires less
10 complex manufacturing steps and equipment.

11 A related general object of the invention is to
12 provide a method which will produce a product which has
13 physical properties as good or better than exhibited by
14 prior art products of a similar kind.

15 A specific object of the invention is to provide a
16 method for manufacturing a superior product which has a
17 lower overall density and specific gravity compared to
18 the prior art, while maintaining all or substantially
19 all of its surface strength, hardness and finish, and
20 moisture resistance. In particular, it is an object to
21 provide an extruded artificial lumber product with
22 similar surface qualities of density, hardness and
23 strength, as the prior art, but having selectively
24 reduced density at its central core. By this means the

1 product of the invention is substantially just as strong
2 as the prior art, but is significantly less dense and
3 more economical to manufacture, and is equal to or
4 superior to the prior art in terms of workability in
5 sawing, drilling, nailing, stapling, and the like.

6 By the method of the present invention, a high-
7 quality wood-like extruded artificial lumber product is
8 produced by finely dividing wood fiber and polyethylene
9 into particles, and then mechanically mixing them
10 together with a measured amount of a powdered
11 endothermic foaming or blowing agent. The resulting
12 feed mix is directly introduced, without pre-
13 pelletization, into a conventional twin-screw extruder
14 where it is compressed and heated into a homogenous
15 plastic state, and then extruded through a molding die
16 to form the structural profile of the desired product.
17 Gases, consisting of vaporized moisture from the
18 feedstock and excess process gas from the foaming agent,
19 is removed by vacuum through passages in the extruder
20 ahead of the molding die. In the process, the carefully
21 controlled amount of foaming agent ingredient has the
22 desirable effect of reducing the density at the center
23 of the extruded profile, while allowing the outer
24 surfaces to remain dense, hard and strong. This has the

1 overall desirable effect of producing a product which is
2 relatively stronger with respect to its density, while
3 continuing to present a smooth, hard well-finished
4 external appearance.

5 It is believed that the controlled amount of
6 foaming agent causes a greater degree of expansion in
7 the center of the extruded profile than at its
8 perimeter, thereby compressing a greater proportion of
9 plastic material against the sides of the extrusion die.
10 This has the effect of increasing the density and
11 strength on the outside of the extrusion, while reducing
12 the density (with no significant loss of overall
13 strength) on the inside. The resulting extruded
14 artificial lumber product can be selectively made with a
15 specific gravity of 1.0, plus or minus 20%, with no
16 significant variation in external dimensions after
17 cooling.

18

19 THE DRAWINGS

20 FIG. 1 is a perspective view of four extruded
21 artificial products, of which one represents a typical
22 prior art product for comparison purposes, and three
23 have been manufactured according to the present
24 invention;

1 FIG. 2 is a schematic diagram of a process
2 embodying the method of the present invention;

3 FIG. 3 is an enlarged horizontal cross-section of
4 the forming die and stabilizing die which receives the
5 molten exudate from the extruder; and

6 FIG. 4 is an enlarged vertical cross-section of the
7 forming die and stabilizing die of Fig. 3.

8

9 DETAILED DESCRIPTION OF THE INVENTION

10 Turning to the drawings, there is shown in Fig. 1 a
11 typical prior art extruded lumber product 10, such as
12 might be manufactured using the process taught in the
13 Laver U.S. Pat. No. 5,516,472. The product 10 might
14 typically be produced in ten foot lengths, with
15 dimensions of 6 inches by 5/4 inches (nominal) and 10,
16 12 or 16 feet in length. This product finds great
17 utility in outdoor benches, tables, and railings, and as
18 deck planking for exterior porches exposed to the
19 weather year-round. Such a prior art product might
20 typically be composed of about two parts finely divided
21 wood fiber and one part finely divided recycled
22 thermoplastic material, along with a lesser amount of
23 thermosetting plastic material. The finely divided
24 ingredients can be mixed directly prior to introduction

1 into an extruder, or they can be pre-pelletized, in the
2 method taught by Deaner, et al. US Patent 5,827,607.
3 Typically, a multiple-stage molding die having a fiber
4 alignment plate or stranding die is used, which aligns
5 the wood fibers, but also cause a high level of back
6 pressure in the extruder.

7 Such prior art artificial lumber planking, while
8 not generally as strong as natural wood, exhibits other
9 favorable qualities. It is generally maintenance free,
10 and can be exposed to the elements indefinitely without
11 significant degradation of either appearance or
12 strength. As for ease of fabrication, it is quite
13 similar to wood in that it can be drilled, sawed, and
14 nailed, and can receive screw and other fasteners, with
15 results very similar to natural wood.

16 However, despite the advantages set forth above,
17 prior art artificial lumber products such as the
18 illustrated example 10 often exhibit deficiencies which
19 can seriously and adversely affect their utility and
20 longevity in certain applications. For example, it has
21 been found that extruded composite products manufactured
22 using the stranding die technology taught in the Laver
23 U.S. Pat. No. 5,516,472 will sometimes suffer from
24 moisture absorption, possibly as a result of having a

1 lower thermoplastic content together with the presence
2 microscopic longitudinal channels created by the forced
3 alignment of the wood fibers during the extrusion
4 process. As a result, the product has, in effect, an
5 "end grain" through which moisture can enter, causing
6 eventually swelling, warping and distortion which can
7 upset the dimensional stability of any structure
8 manufactured with these materials.

9 In addition, while the prior art extruded
10 artificial lumber products 10 generally have a superior
11 surface in terms of strength, hardness and appearance,
12 they are generally quite dense, with some having
13 specific gravities substantially higher than 1.0,
14 meaning that they consume more raw materials per board
15 foot of product, and have a poorer strength-to-weight
16 ratio in comparison to natural wood. They will not
17 float at all.

18 Finally, the manufacture of prior art artificial
19 lumber products 10 by the prior art methods described
20 above is relatively costly and time-consuming because of
21 the need for either pre-pelletization or a pre-melt step
22 in some cases, and for multiple-part extrusion dies
23 (including stranding dies) in others.

1 Referring again to the drawings, there are also
2 shown in Fig. 1 three additional extruded artificial
3 lumber sections 12, 14 and 16, in the form of deck
4 planks, manufactured according to the present invention.
5 Improved plank 12 exhibits the same hard, strong, smooth
6 surface as prior art plank 10, but has at its center a
7 region 13 of reduced density which lowers the overall
8 density and weight of the plank without significantly
9 affecting its strength. Even though the density
10 reduction may reduce the tensile strength and modulus of
11 the product at its center, the fact that the outer
12 surfaces are effectively unaffected causes the overall
13 strength and modulus of the product to be substantially
14 unchanged.

15 The density reduction of plank 12 at its center 13
16 is achieved by the addition of a controlled quantity of
17 foaming agent, preferably up to 1% of an endothermic
18 foaming agent such as bicarbonate of soda. This agent
19 is added and mixed into the wood fiber and thermoplastic
20 polymer components which, together with small quantities
21 of certain other components, comprise the feedstock of
22 the extruder. It has been found that it is possible to
23 control the expansion of the foaming agent in a way
24 which substantially confines it to the center of the

1 extruded product, thereby achieving additional lightness
2 without any sacrifice in surface characteristics or
3 overall strength.

4 The amount of endothermic foaming agent in the
5 feedstock mix has been found to be relatively critical.
6 Referring again to Fig. 1, plank 14 exhibits bowed outer
7 surfaces because of excessive expansion at its center
8 15. Similarly, the center 17 of plank 16 has not
9 expanded sufficiently, or has even shrunk after leaving
10 the extruder, giving the cross-section a "dog bone"
11 shape which is also unacceptable. It is therefore
12 important to adjust and balance the concentration of
13 endothermic foaming agent against the wood fiber and
14 thermoplastic polymer components of the feedstock
15 mixture so that a plank 12 with dimensionally stable
16 surfaces is achieved, and not a bowed plank 14 or sunken
17 plank 16 which may possess a reduced density at its
18 center, but which may be dimensionally unacceptable.

19 Turning to Fig. 2, there is shown in schematic form
20 a production line for producing the improved,
21 dimensionally stable plank 12 of the present invention.
22 A supply of wood fiber or other fibrous cellulosic
23 material 18 is introduced into a pulverizer or shredder
24 19 where it is finely divided into particles having a

1 maximum length dimension generally no smaller than 80
2 mesh (about 0.007 inches), and no larger than about 0.5
3 inches, with the preferred range being 10-40 mesh.

4 Another supply of thermoplastic material 20, which is
5 preferably scrap polyethylene such as may be reclaimed
6 from a materials recycling program, is similarly finely
7 divided in a pulverizer or shredder 21 into particles
8 generally no smaller than 80 mesh, with the preferred
9 range being 10-60 mesh.

10 After pulverization, the finely divided wood fiber
11 and thermoplastic particles are conveyed, such as by air
12 conveyor, to a mixer 22. To the mixer 22 is also added
13 a quantity of powdered endothermic foaming agent 23 such
14 as bicarbonate of soda, and (if desired) up to about 1%
15 of a wax lubricant 24.

16 In practice, the following ranges (parts by weight)
17 of components have been found most desirable in
18 achieving the objects of the invention:

		Wood Fiber	Polymer	Foaming Agent	Lubricant
19					
20					
21	Composition A	50	50	0.6	0.8
22	Composition B	60	40	0.3	1.0
23	Composition C	40	60	0.7	0.6

1 If desired, up to 5 parts of a thermoplastic olefin
2 can also be added for optimizing melt flow
3 characteristics.

4 According to the invention, the wood fiber,
5 thermoplastic and foaming agent ingredients are
6 thoroughly mixed in the mixer 22 and then conveyed, by
7 means such as an air conveyor, to the input hopper 25 of
8 a screw-type extruder 26. Excellent results have been
9 achieved using the commercially available Cincinnati
10 Milacron CM-80-BP twin screw extruder driven by motor
11 27. As is well known in the art, the twin screw
12 extruder uses meshed counter-rotating flights (not
13 shown) which have a larger pitch at the inlet end and a
14 smaller pitch at the output end. The flights are heated
15 internally, and the extruder barrel is also heated.

16 In combination, the heat imparted to the feedstock
17 mixture by the heated extruder flights and barrel, plus
18 the mechanical shearing and compression caused by the
19 differential pitch of the flights, cause the feedstock
20 mixture temperature to be raised to a point where it
21 becomes plastic and homogenous, with the wood fibers
22 being intimately mixed, coated and bound in the melted
23 thermoplastic. In addition, any residual moisture in
24 the feedstock components is vaporized, and as the

1 mixture heats further, its temperature is desirably in
2 the range of 320° F to 400° F, which causes the
3 endothermic foaming agent to become activated, absorbing
4 some of the heat energy and releasing carbon dioxide
5 gas.

6 As the heated and compressed feedstock approaches
7 the extruder die 29 at the exit end of the extruder,
8 excess volatiles including vaporized moisture and excess
9 foaming agent gas (principally carbon dioxide) are
10 removed from the extruder ahead of the molding die by a
11 vacuum pump 28. In practice, it has been found that the
12 best results are obtained at vacuum levels of at least
13 25 inches of mercury, with the best operating range
14 being between 27 and 30 inches of mercury. With less
15 vacuum, the resulting product is more sensitive to
16 moisture, possibly because the remaining volatiles
17 (water and carbon dioxide) permeating the melt and
18 create fissures in the final product, into which water
19 may penetrate. On the other hand, vacuum levels of 30
20 inches of mercury and more tend to negate the effect of
21 the foaming agent, leading to insufficient density
22 reduction, insufficient dimensional stability on leaving
23 the extruder, and poor workability in the finished
24 product.

1 With the process of the present invention, no
2 special multiple die sets, and no fiber alignment or
3 stranding die, are needed to produce a strong, stable,
4 moisture-resistant product. As shown in Figs. 3 and 4;
5 the extrusion die 29 has a converging entrance 33
6 leading to a throat 34 sized to produce the desired
7 degree of pressure drop leaving the extruder, and a
8 diverging exit 35 passage allowing for expansion of the
9 melt in cross-section to form the desired profile of the
10 extruded product.

11 From the exit passage the extruded product passes
12 through a stabilization die 36 where it cools
13 sufficiently to retain its shape upon entering the spray
14 chamber 30. In practice, the extruded material leaving
15 the throat of the die expands just sufficiently to take
16 the fill the exit passage and thereby take its final
17 shape, without undue pulling or dragging across its
18 surface which might cause fissures known as "melt
19 fractures".

20 From the extruder 26 and die 29, the formed ribbon
21 of extruded product passes into a spray chamber 30 where
22 it is cooled by spray jets of water from a reservoir 31
23 as is well understood in the art. Once cooled, it
24 passes by conventional means to a cutoff station 32

1 where a traveling table or "flying" cutoff knife or saw
2 cuts the product to any length desired.

3 A typical product manufactured by the method of the
4 invention has been found to exhibit the following
5 characteristics (typical values):

6	Modulus of elasticity	285,758 psi	ASTM D4761
7	Modulus of rupture	1676 psi	ASTM D4761
8	Tensile strength	786 psi	ASTM D198
9	Shear strength	706 psi	ASTM D143
10	Screw withdrawal force	650 lb/in	ASTM D1761
11	Nail withdrawal force	177 lb/in	ASTM D1761
12	Coefficient of thermal expansion	4.5×10^{-5}	ASTM E228
13	Water absorption	1.66%	ASTM D1037
14	Density (S.G.)	1.0	

1 I CLAIM AS MY INVENTION:

2 1. A process for manufacturing a composite
3 extruded structural product having a desired profile
4 from thermoplastic material and wood fiber comprising
5 the steps of:
6 finely dividing the thermoplastic material and wood
7 fiber each into particles no smaller than about 0.007
8 inches and no larger than about 0.5 inches in length;
9 mechanically mixing together the thermoplastic
10 particles and the wood fiber particles in a ratio of
11 between 60%-40% and 40%-60% by weight, together with an
12 effective amount of a foaming agent, to form a feedstock
13 mixture;
14 introducing the feedstock mixture, without pre-
15 pelletization, into a screw-type extruder;
16 mechanically mixing, compressing and heating said
17 feedstock mixture in said extruder until it becomes
18 plastic and homogenous;
19 extruding said heated, plastic, homogenous mixture
20 through a molding die into the structural profile of a
21 desired product;
22 cooling said extruded product upon emerging from
23 said molding die; and

1 cutting the cooled extruded product into desired
2 lengths.

3 2. The process of claim 1 in which an effective
4 amount of foaming agent ingredient is selected to create
5 an extruded product having a specific gravity of between
6 about 0.8 and about 1.2 with no significant dimensional
7 variation after cooling.

8 3. The process of claim 1 in which the effective
9 amount of foaming agent ingredient is up to about 1% by
10 weight.

11 4. The process of claim 1 in which the foaming
12 agent ingredient is an endothermic foaming agent.

13 5. The process of claim 1 in which the foaming
14 agent ingredient is bicarbonate of soda.

15 6. The process of claim 1 including the
16 additional step of extracting excess volatiles under
17 vacuum from said extruder, thereby producing an extruded
18 product having a surface which is relatively dense,
19 tight-grained and strong, and a center which is
20 relatively more porous and less dense.

21 7. The process of claim 6 in which the vacuum
22 extraction step is performed using a vacuum of at least
23 25 inches of mercury.

1 8. The process of claim 1 in which up to 1% by
2 weight of wax lubricant is mixed into the feedstock
3 mixture prior to introduction into the extruder.

4 9. The process of claim 1 in which up to 5% by
5 weight of thermoplastic olefin is mixed into the
6 feedstock mixture prior to introduction into the
7 extruder.

8 10. The process of claim 1 in which the molding
9 die has a converging entrance, a throat, and a diverging
10 exit terminating in the profile of the desired
11 structural product.

12 11. The process of claim 1 in which the extruded
13 product upon emerging from said molding die is cooled
14 with a direct water spray, and said cooled extruded
15 product is cut into desired lengths with a traveling
16 saw.

17 12. A process for manufacturing a composite
18 extruded structural product having a desired profile
19 from recycled polyethylene and wood fiber comprising the
20 steps of:

21 finely dividing recycled polyethylene and wood
22 fiber each into particles of a size between 10 mesh and
23 40 mesh;

1 mechanically mixing together the polyethylene
2 particles and the wood fiber particles in a ratio of
3 between 60%-40% and 40%-60% by weight, and an effective
4 amount of a powdered endothermic foaming agent, to form
5 a feedstock mixture;
6 introducing the feedstock mixture, without pre-
7 pelletization, into a heated screw-type extruder
8 discharging into a molding die, said molding die having
9 an entrance, a throat, and an exit having the shape of a
10 desired product;
11 mechanically mixing, compressing and heating said
12 feedstock mixture in said extruder until it becomes
13 plastic and homogenous;
14 extracting excess volatiles and foaming agent
15 process gas under vacuum from said feedstock mixture
16 prior to entering said molding die;
17 forcing said heated, plastic, homogenous mixture
18 through said molding die to produce an extruded product
19 having a surface which is relatively dense, tight-
20 grained and strong, and a center which is relatively
21 more porous and less dense;
22 cooling said extruded product upon emerging from
23 said molding die; and

1 cutting the cooled extruded product into desired
2 lengths.

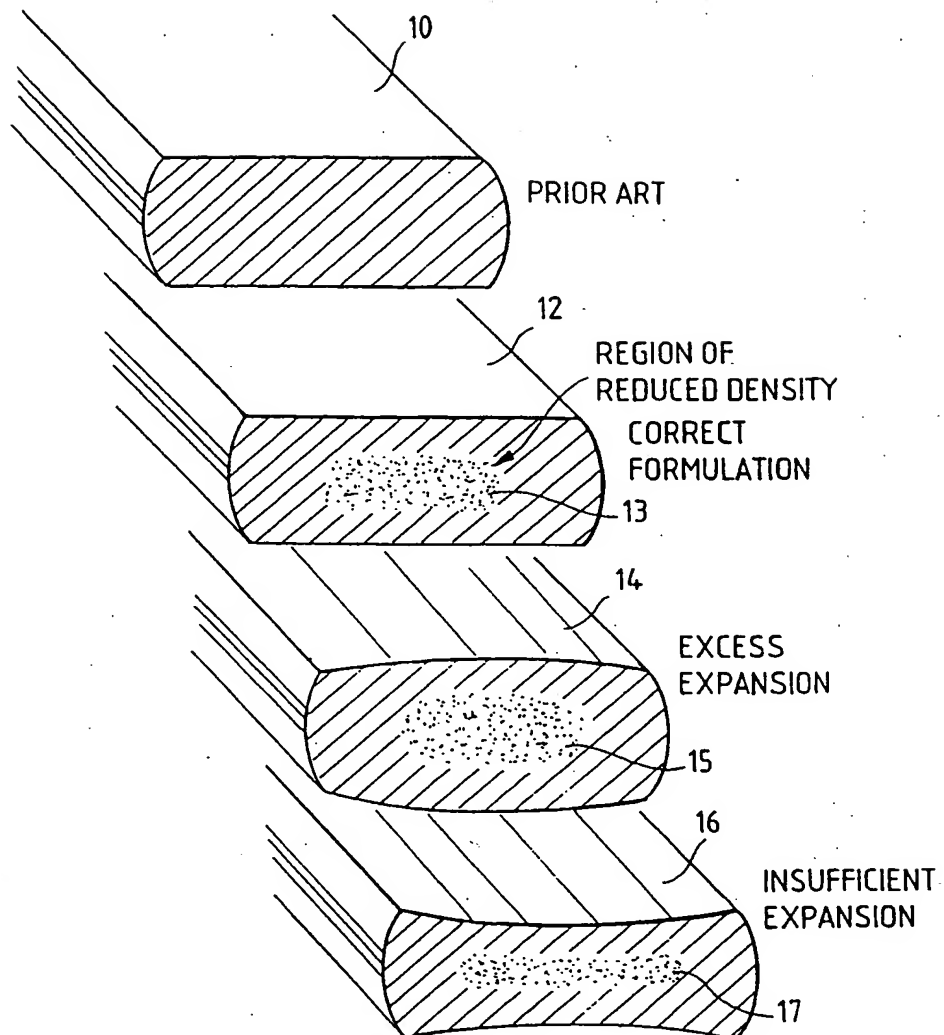
3 13. A composite extruded artificial lumber product
4 having a surface which is relatively dense, tight-
5 grained and strong, and a center which is relatively
6 more porous and less dense, manufactured by the process
7 of claim 1.

8 14. A composite extruded artificial lumber product
9 having a surface which is relatively dense, tight-
10 grained and strong, and a center which is relatively
11 more porous and less dense, manufactured by the process
12 of claim 12.

13 15. The composite extruded artificial lumber
14 product of claim 13 having a specific gravity between
15 about 0.8 and about 1.2 with no significant dimensional
16 variation after cooling.

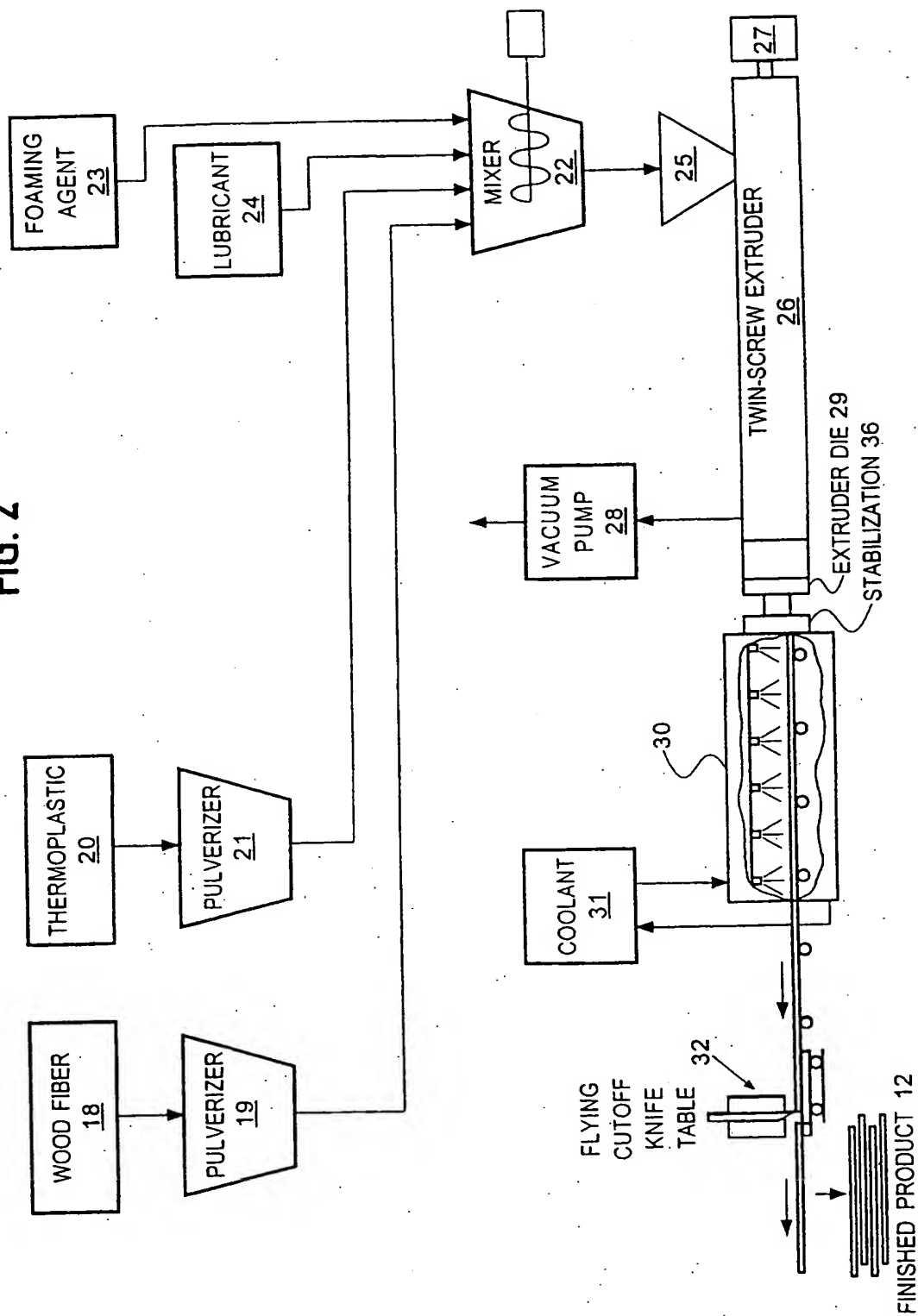
17 16. The composite extruded artificial lumber
18 product of claim 14 having a specific gravity between
19 about 0.8 and about 1.2 with no significant dimensional
20 variation after cooling.

FIG. 1



2/3

FIG. 2



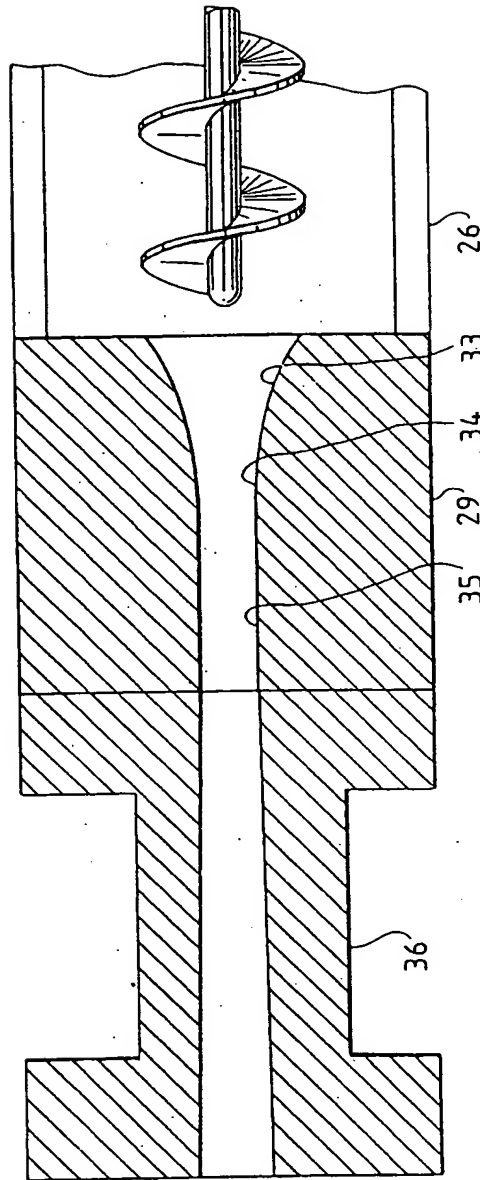


FIG. 3

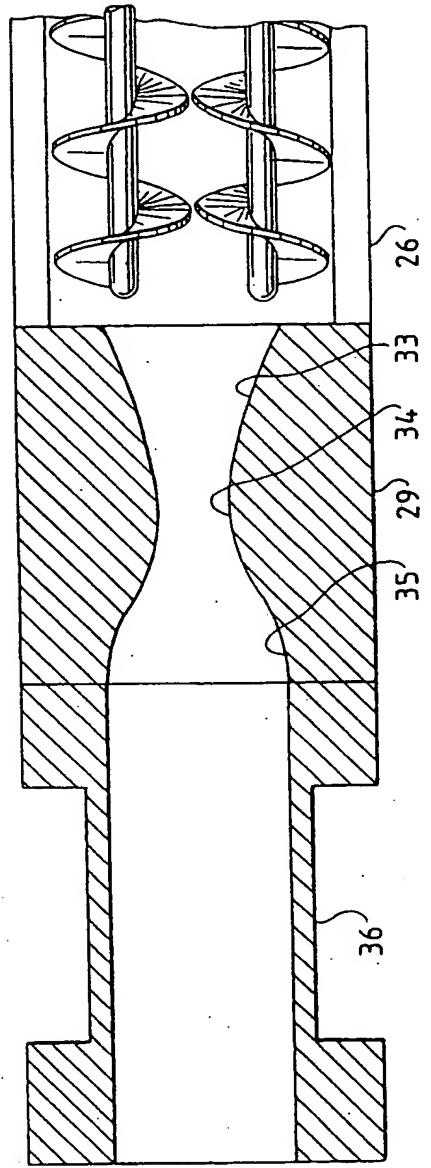


FIG. 4

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/02345

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) :B29C 47/78, 47/36

US CL : 264/118,122,913,920; 428/903.3

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 264/118,122,913,920; 428/903.3; 425/382R, 382.4

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WEST 2.0 search terms, see claim 1

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5,516,472 A (LAVER et al) 1A May 1996	1-16

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

Special categories of cited documents:	
* "A"	document defining the general state of the art which is not considered to be of particular relevance
* "E"	earlier document published on or after the international filing date
* "L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
* "O"	document referring to an oral disclosure, use, exhibition or other means
* "P"	document published prior to the international filing date but later than the priority date claimed
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Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231

Facsimile No. (703) 305-3230

Authorized officer

RICH WEISBERGER

Telephone No. (703) 308-2351